Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(2-Hydroxy-3-methylbenzylideneamino)-2,3-dimethyl-1-phenyl-1*H*-pyrazol-5(2*H*)-one

Yun-Fa Zheng* and Ming-Hua Yang

Department of Chemistry, Lishui University, 323000 Lishui, Zhejiang, People's Republic of China
Correspondence e-mail: zjlsxyhx@126.com

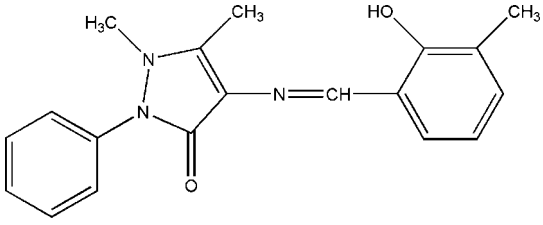
Received 26 June 2007; accepted 1 July 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.033; wR factor = 0.063; data-to-parameter ratio = 12.9.

In the title Schiff base, $C_{19}H_{19}N_3O_2$, the dihydropyrazole ring is essentially planar, with an r.m.s. deviation of 0.0289 Å for the fitted atoms. This ring makes dihedral angles of 41.8 (2) and 51.1 (3)° with the phenyl and benzene rings, respectively. In the crystal structure, there is one intramolecular O—H···N hydrogen bond.

Related literature

For related literature, see: Alemi & Shaabani (2000); Alizadeh *et al.* (1999); Allen (2002); Bruno *et al.* (2004); Jin *et al.* (2004); Johnson *et al.* (1996); Kim & Shin (1999); Wang & Zheng (2007); Yan *et al.* (2007).



Experimental

Crystal data

$C_{19}H_{19}N_3O_2$
M_r = 321.37
Monoclinic, $P2_1/n$
a = 7.5655 (19) Å
b = 7.5713 (19) Å
c = 29.146 (7) Å
β = 92.006 (4)°
V = 1668.5 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.34 × 0.28 × 0.17 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.986
9793 measured reflections
2820 independent reflections
1464 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.063
S = 0.88
2820 reflections
219 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···N1	0.82	1.87	2.596 (2)	148

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Science Planning Technical Foundation of Zhejiang Province (grant No. 2006 C31067) and the Research Foundation of Lishui University for financial support

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2162).

References

Alemi, A. A. & Shaabani, B. (2000). *Acta Chim. Slov.* **47**, 363–369.
Alizadeh, N., Ershad, S., Naeimi, H., Sharghi, H. & Shamsipur, M. (1999). *Pol. J. Chem.* **73**, 915–925.
Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1998). *SMART* (Version 5.0) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Version 6.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Kessler, M., Luo, J., Motherwell, W. D. S., Purkis, L. H., Smith, B. R., Taylor, R., Cooper, R. I., Harris, S. E. & Orpen, A. G. (2004). *J. Chem. Inf. Comput. Sci.* **44**, 2133–2144.
Jin, Z.-M., Li, L., Li, M.-C., Hu, M.-L. & Shen, L. (2004). *Acta Cryst.* C**60**, o642–o643.
Johnson, C. P., Atwood, J. L., Steed, J. W., Bauer, C. B. & Rogers, R. D. (1996). *Inorg. Chem.* **35**, 2602–2610.
Kim, G. J. & Shin, J. W. (1999). *Catal. Lett.* **63**, 83–89.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wang, L.-G. & Zheng, Y.-F. (2007). *Acta Cryst.* E**63**, m390–m391.
Yan, G.-B., Zhang, C.-N. & Yang, M.-H. (2007). *Acta Cryst.* E**63**, o2663–o2664.

 doi:10.1107/S1600536807032060

supplementary materials

Acta Cryst. (2007). E**63**, o3421 [**doi:10.1107/S1600536807032060**]

4-(2-Hydroxy-3-methylbenzylideneamino)-2,3-dimethyl-1-phenyl-1*H*-pyrazol-5(2*H*)-one

Y.-F. Zheng and M.-H. Yang

Comment

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes, (Johnson *et al.*, 1996; Alizadeh *et al.*, 1999; Wang & Zheng, 2007). Schiff bases that have solvent-dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optically active) materials (Alemi & Shaabani, 2000). They are also useful in the asymmetric oxidation of methyl phenyl sulfide and are enantioselective (Kim & Shin, 1999). In this paper, we report the synthesis and crystal structure of the title compound.

The molecular structure of the title compound, $C_{19}H_{19}N_3O_2$, (Fig. 1), is very similar to the compound, $C_{19}H_{19}N_3OS$, that was reported recently by Yan and his co-workers (Yan *et al.*, 2007). In the latter, there are intermolecular C—H···O hydrogen bonds, leading to a chain parallel to the *a* axis. In contrast, the crystal structure of the title compound has only an intramolecular O—H···N hydrogen bond (Table 1).

The C12—N1 bond length is 1.29 (9) Å, indicative of a C═N double bond. The other C—N, C—O and C—C distances show no remarkable features (Cambridge Structural Database, August 2006 version; Allen, 2002; Mogul, version 1.1; Bruno *et al.*, 2004) The dihydropyrazole ring (C7–C9/N2,N3) is essentially planar with an r.m.s. deviation of 0.0289 Å for the fitted atoms; this ring makes a dihedral angle of 41.8 (2)° and 51.1 (3)° with the phenyl and benzene rings, respectively. Bond conjugation is observed in the (N1/C12/C13) sequence of atoms (Jin *et al.*, 2004).

Experimental

Under nitrogen, a mixture of 4-amino-2,3-dimethyl-1-phenyl-1,2-dihydropyrazol-5-one (1.45 g,10 mmol), Na_2SO_4 (3.0 g) and 2-hydroxy-3-methylbenzadehyde (1.60 g, 10 mmol) in absolute ethanol (20 ml) was refluxed for about 12 h to yield a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2 *x* 15 ml) and brine (8 ml). After drying over Na_2SO_4, the solvent was removed under vacuum, and a yellow solid was isolated in 92% yield (3.1 g). Colourless single crystals of the title Schiff base suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol (4:1) by slow evaporation of the solvents at room temperature over a period of about one week.

Refinement

All H atoms were placed in calculated positions (C—H = 0.93 (aromatic) or 0.96 Å (methyl), O—H = 0.82 Å) and refined using a riding model, with U_{iso}(H) = xU_{eq}(carrier atom), where *x* = 1.5 for methyl and 1.2 for other H atoms.

Figures

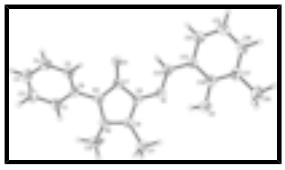


Fig. 1. The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

4-(2-Hydroxy-3-methylbenzylideneamino)-2,3-dimethyl-1-phenyl-1*H*-pyrazol- 5(2*H*)-one

Crystal data

$C_{19}H_{19}N_3O_2$	F_{000} = 680
M_r = 321.37	D_x = 1.279 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2820 reflections
a = 7.5655 (19) Å	θ = 2.8–25.1º
b = 7.5713 (19) Å	μ = 0.09 mm^{-1}
c = 29.146 (7) Å	T = 298 (2) K
β = 92.006 (4)º	Block, colourless
V = 1668.5 (7) Å^3	0.34 × 0.28 × 0.17 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	2820 independent reflections
Radiation source: fine-focus sealed tube	1464 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.039
T = 298(2) K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −9→9
T_{min} = 0.972, T_{max} = 0.986	k = −9→8
9793 measured reflections	l = −32→34

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0105P)^2 + 0.28P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.063	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.88	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
2820 reflections	$\Delta\rho_{min}$ = −0.12 e Å^{-3}

219 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0221 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.08050 (14)	0.08844 (16)	0.90981 (4)	0.0604 (4)
N1	0.84312 (18)	0.21964 (18)	0.98889 (5)	0.0519 (4)
O2	0.74420 (14)	0.3600 (2)	1.06511 (4)	0.0747 (4)
H2	0.7338	0.3083	1.0404	0.112*
N3	0.80598 (16)	0.04599 (19)	0.87312 (5)	0.0514 (4)
C7	0.9180 (2)	0.0935 (2)	0.91056 (6)	0.0487 (5)
N2	0.62994 (16)	0.0906 (2)	0.88302 (5)	0.0561 (4)
C9	0.6328 (2)	0.1404 (2)	0.92814 (6)	0.0535 (5)
C8	0.8009 (2)	0.1501 (2)	0.94558 (6)	0.0478 (5)
C17	0.9636 (3)	0.4604 (2)	1.11831 (7)	0.0597 (5)
C13	1.0482 (2)	0.3261 (2)	1.04663 (6)	0.0487 (5)
C12	1.0043 (2)	0.2453 (2)	1.00277 (6)	0.0528 (5)
H14	1.0952	0.2108	0.9841	0.063*
C1	0.7648 (2)	0.1442 (2)	0.79366 (7)	0.0610 (5)
H1	0.6630	0.2052	0.8011	0.073*
C19	0.9186 (2)	0.3813 (2)	1.07651 (6)	0.0534 (5)
C14	1.2249 (2)	0.3531 (2)	1.05965 (7)	0.0616 (5)
H16	1.3127	0.3181	1.0400	0.074*
C6	0.8584 (2)	0.0497 (2)	0.82697 (6)	0.0504 (5)
C15	1.2716 (3)	0.4302 (3)	1.10085 (7)	0.0688 (6)
H17	1.3901	0.4477	1.1092	0.083*
C5	1.0092 (2)	−0.0415 (2)	0.81610 (7)	0.0627 (6)
H5	1.0710	−0.1072	0.8383	0.075*
C16	1.1401 (3)	0.4819 (2)	1.12984 (7)	0.0675 (6)
H18	1.1720	0.5326	1.1580	0.081*
C2	0.8231 (3)	0.1476 (3)	0.74941 (7)	0.0770 (6)
H2A	0.7597	0.2103	0.7269	0.092*
C10	0.4910 (2)	−0.0260 (3)	0.86365 (6)	0.0729 (6)

H10A	0.4995	−0.1397	0.8782	0.109*
H10B	0.5057	−0.0390	0.8312	0.109*
H10C	0.3771	0.0245	0.8689	0.109*
C11	0.4661 (2)	0.1818 (3)	0.95186 (6)	0.0783 (7)
H11A	0.3910	0.0796	0.9514	0.117*
H11B	0.4062	0.2777	0.9364	0.117*
H11C	0.4939	0.2148	0.9831	0.117*
C4	1.0673 (3)	−0.0339 (3)	0.77143 (8)	0.0809 (7)
H4	1.1704	−0.0924	0.7639	0.097*
C3	0.9731 (3)	0.0598 (3)	0.73841 (7)	0.0856 (7)
H3	1.0118	0.0632	0.7085	0.103*
C18	0.8224 (3)	0.5192 (3)	1.14998 (7)	0.0898 (7)
H18A	0.8763	0.5584	1.1785	0.135*
H18B	0.7443	0.4222	1.1556	0.135*
H18C	0.7563	0.6146	1.1360	0.135*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0383 (7)	0.0868 (10)	0.0559 (9)	0.0026 (7)	−0.0011 (6)	−0.0061 (7)
N1	0.0470 (9)	0.0637 (11)	0.0449 (11)	0.0013 (8)	−0.0014 (8)	0.0021 (8)
O2	0.0535 (8)	0.1054 (12)	0.0656 (11)	−0.0024 (8)	0.0083 (7)	−0.0188 (9)
N3	0.0375 (8)	0.0723 (11)	0.0443 (11)	0.0028 (8)	−0.0002 (7)	−0.0027 (8)
C7	0.0435 (11)	0.0596 (13)	0.0427 (12)	0.0000 (9)	−0.0037 (9)	0.0042 (9)
N2	0.0362 (8)	0.0804 (12)	0.0515 (11)	−0.0010 (8)	−0.0025 (7)	−0.0059 (9)
C9	0.0435 (11)	0.0712 (14)	0.0458 (13)	0.0027 (10)	0.0041 (9)	−0.0014 (10)
C8	0.0415 (11)	0.0601 (13)	0.0417 (13)	0.0004 (9)	0.0002 (9)	0.0020 (10)
C17	0.0706 (14)	0.0623 (14)	0.0465 (14)	−0.0018 (11)	0.0083 (11)	−0.0014 (11)
C13	0.0492 (11)	0.0546 (12)	0.0421 (13)	0.0008 (9)	−0.0003 (10)	0.0003 (9)
C12	0.0488 (11)	0.0602 (13)	0.0496 (13)	0.0036 (9)	0.0046 (9)	0.0009 (10)
C1	0.0650 (13)	0.0713 (14)	0.0461 (14)	0.0038 (11)	−0.0034 (11)	−0.0037 (11)
C19	0.0524 (12)	0.0605 (13)	0.0474 (13)	−0.0026 (10)	0.0021 (10)	0.0013 (10)
C14	0.0537 (12)	0.0725 (14)	0.0583 (15)	0.0012 (11)	−0.0034 (10)	−0.0061 (11)
C6	0.0483 (11)	0.0612 (13)	0.0417 (13)	−0.0036 (10)	0.0009 (9)	−0.0042 (10)
C15	0.0636 (13)	0.0725 (15)	0.0694 (16)	−0.0046 (12)	−0.0116 (12)	−0.0056 (12)
C5	0.0533 (12)	0.0781 (15)	0.0567 (15)	0.0030 (11)	0.0015 (10)	−0.0079 (12)
C16	0.0856 (15)	0.0650 (14)	0.0512 (14)	−0.0054 (12)	−0.0083 (12)	−0.0066 (11)
C2	0.0888 (16)	0.0906 (17)	0.0511 (17)	−0.0013 (14)	−0.0041 (13)	0.0049 (12)
C10	0.0476 (11)	0.0952 (16)	0.0749 (15)	−0.0097 (11)	−0.0093 (10)	−0.0093 (12)
C11	0.0466 (12)	0.1223 (19)	0.0664 (15)	0.0070 (12)	0.0074 (10)	−0.0118 (13)
C4	0.0611 (14)	0.115 (2)	0.0670 (17)	0.0036 (13)	0.0129 (13)	−0.0166 (15)
C3	0.0867 (17)	0.121 (2)	0.0496 (16)	−0.0080 (16)	0.0126 (13)	−0.0039 (14)
C18	0.0988 (17)	0.1070 (19)	0.0649 (16)	−0.0019 (14)	0.0202 (13)	−0.0184 (13)

Geometric parameters (Å, °)

O1—C7	1.2308 (17)	C14—C15	1.371 (2)
N1—C12	1.2858 (19)	C14—H16	0.9300
N1—C8	1.3942 (19)	C6—C5	1.380 (2)

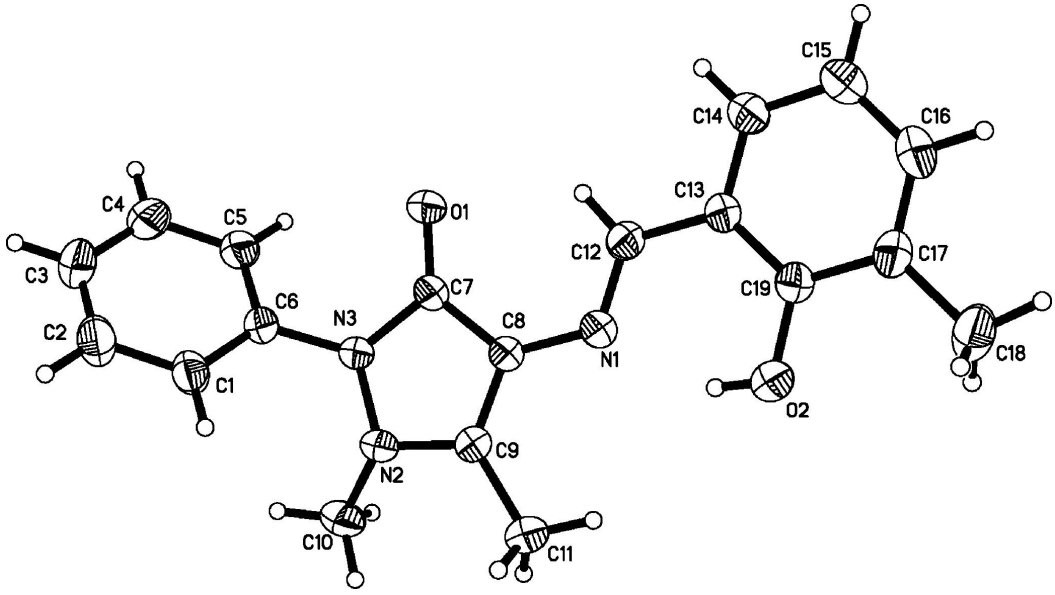
O2—C19	1.3588 (18)	C15—C16	1.384 (2)
O2—H2	0.8200	C15—H17	0.9300
N3—C7	1.4050 (19)	C5—C4	1.390 (2)
N3—N2	1.4136 (16)	C5—H5	0.9300
N3—C6	1.416 (2)	C16—H18	0.9300
C7—C8	1.440 (2)	C2—C3	1.363 (3)
N2—C9	1.368 (2)	C2—H2A	0.9300
N2—C10	1.470 (2)	C10—H10A	0.9600
C9—C8	1.355 (2)	C10—H10B	0.9600
C9—C11	1.493 (2)	C10—H10C	0.9600
C17—C16	1.375 (2)	C11—H11A	0.9600
C17—C19	1.389 (2)	C11—H11B	0.9600
C17—C18	1.504 (2)	C11—H11C	0.9600
C13—C14	1.392 (2)	C4—C3	1.374 (3)
C13—C19	1.398 (2)	C4—H4	0.9300
C13—C12	1.445 (2)	C3—H3	0.9300
C12—H14	0.9300	C18—H18A	0.9600
C1—C2	1.378 (2)	C18—H18B	0.9600
C1—C6	1.381 (2)	C18—H18C	0.9600
C1—H1	0.9300		
C12—N1—C8	121.75 (15)	C1—C6—N3	121.64 (17)
C19—O2—H2	109.5	C14—C15—C16	119.08 (18)
C7—N3—N2	109.11 (13)	C14—C15—H17	120.5
C7—N3—C6	123.46 (14)	C16—C15—H17	120.5
N2—N3—C6	119.10 (14)	C6—C5—C4	119.04 (19)
O1—C7—N3	123.64 (16)	C6—C5—H5	120.5
O1—C7—C8	131.41 (16)	C4—C5—H5	120.5
N3—C7—C8	104.92 (14)	C17—C16—C15	122.01 (18)
C9—N2—N3	106.19 (13)	C17—C16—H18	119.0
C9—N2—C10	121.46 (15)	C15—C16—H18	119.0
N3—N2—C10	116.36 (14)	C3—C2—C1	120.5 (2)
C8—C9—N2	111.03 (15)	C3—C2—H2A	119.7
C8—C9—C11	127.75 (18)	C1—C2—H2A	119.7
N2—C9—C11	121.19 (15)	N2—C10—H10A	109.5
C9—C8—N1	122.83 (16)	N2—C10—H10B	109.5
C9—C8—C7	108.10 (16)	H10A—C10—H10B	109.5
N1—C8—C7	128.84 (15)	N2—C10—H10C	109.5
C16—C17—C19	118.14 (18)	H10A—C10—H10C	109.5
C16—C17—C18	121.31 (19)	H10B—C10—H10C	109.5
C19—C17—C18	120.55 (18)	C9—C11—H11A	109.5
C14—C13—C19	118.29 (17)	C9—C11—H11B	109.5
C14—C13—C12	119.47 (16)	H11A—C11—H11B	109.5
C19—C13—C12	122.23 (16)	C9—C11—H11C	109.5
N1—C12—C13	121.88 (16)	H11A—C11—H11C	109.5
N1—C12—H14	119.1	H11B—C11—H11C	109.5
C13—C12—H14	119.1	C3—C4—C5	120.26 (19)
C2—C1—C6	119.64 (18)	C3—C4—H4	119.9
C2—C1—H1	120.2	C5—C4—H4	119.9
C6—C1—H1	120.2	C2—C3—C4	120.2 (2)

O2—C19—C17	118.16 (16)	C2—C3—H3	119.9
O2—C19—C13	120.54 (16)	C4—C3—H3	119.9
C17—C19—C13	121.30 (17)	C17—C18—H18A	109.5
C15—C14—C13	121.16 (18)	C17—C18—H18B	109.5
C15—C14—H16	119.4	H18A—C18—H18B	109.5
C13—C14—H16	119.4	C17—C18—H18C	109.5
C5—C6—C1	120.36 (18)	H18A—C18—H18C	109.5
C5—C6—N3	117.99 (17)	H18B—C18—H18C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N1	0.82	1.87	2.596 (2)	148

Fig. 1


C15
C14
C16
C13
C4
C5
O1
C12
C17
C3
C7
C19
C6
N3
C8
C2
N1
C18
C1
O2
C9
N2
C10
C11